



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of Kazuyoshi Ichihara et al.

Serial No.: 09/700,494

Group Art Unit: 1621

Filed: 11/16/2000

Examiner: Paul A. Zucker

For: Process for Producing Fluoroalkylcarboxylic Acid

DECLARATION

Honorable Commissioner of Patents and Trademarks

Washington, D.C. 20231

Sir :

I, Kazuyoshi Ichihara, hereby declare:

1) That I am one of the inventors of the instant invention, and

2) That the experiment given below was carried out under my general direction and supervision.

1) Comparative Experiment 1

$\text{H}(\text{CF}_2)_6\text{CH}_2\text{OH}$  (66.40 g, 0.20 moles), 55% nitric acid (68.7 g, 0.60 moles) and  $\text{FeCl}_2 \cdot n\text{H}_2\text{O}$  (0.0010 g) were placed

into an autoclave containing a 200-ml SUS316 (stainless-steel) pressure vessel having stirring blades, a thermometer protection tube and an insert tube, and equipped with a pressure gauge, a safety valve and a supply line from an oxygen bomb. Oxygen was fed into the autoclave until the pressure reached 0.3 MPa gauge. Then the mixture was heated at 125°C with stirring for 9 hours.  $\text{H}(\text{CF}_2)_6\text{CH}_2\text{OH}$  did not react at all and was recovered unchanged.

## 2) Consideration

In the reaction for oxidation of fluoroalkyl alcohol with nitric acid, if oxygen is fed at the beginning of the reaction as in Comparative Experiment 1, the reaction does not proceed. In contrast, if oxygen is fed not at the beginning, but in the course of the reaction as in the Examples of the specification of this application, the required amount of nitric acid can be reduced to a stoichiometric amount or less relative to the fluoroalkyl alcohol and the reaction pressure can also be controlled.

I, the undersigned, declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 7/14/2003

Kazuyoshi Ichihara

Kazuyoshi Ichihara